

BENCH SCALE TESTING PLAN:
TREATABILITY STUDY OF PERMEABLE
REACTIVE BARRIERS FOR
REMEDIATION OF ROCKY FLATS
ENVIRONMENTAL TECHNOLOGY SITE
GROUNDWATER

Prepared for

Rocky Mountain Remediation Services L L C

Prepared by

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1.0 INTRODUCTION

This test plan, prepared for Rocky Mountain Remediation Services (RMRS), presents the scope of work for the bench-scale evaluation of permeable barrier materials to treat dissolved organic compounds present in groundwater at the Rocky Flats Environmental Technology Site This work plan incorporates the test parameters outlined in the scope of work (SOW) prepared by RMRS

The remainder of the first section of this work plan provides background information on the technology and its potential application to the RFETS site. The second section describes the objectives and scope of the column tests which form the core of the study. Section 3 describes test methodology and means of interpreting column test data. The topics to be covered in our report are presented in Section 4, and a project schedule is presented in Section 5.

1.1 Treatment Technology Description

As a consequence of the significant limitations of pump-and-treat systems, in situ permeable reaction barriers have been identified as an innovative alternative groundwater remediation technology (Gillham, 1996). The concept is remarkably simple, involving the construction of a permeable wall or barrier containing appropriate reactive materials across the path of a contaminant plume. As the contaminated groundwater passes through the wall, the contaminants are removed through chemical or physical processes. Various configurations of these in-situ treatment systems have been evaluated, based on site-specific conditions. Particular advantages that one would hope to achieve from in situ reactive barriers include.

- conservation of groundwater resources
- long-term passive treatment
- absence of waste materials requiring treatment or disposal
- absence of invasive surface structures and equipment
- reduced capital cost

Several different types of media have been suggested for use in these in-situ treatment zones. The most advanced stage of application has been achieved with metal based systems to remove

both chlorinated organic compounds and trace metals

Under highly reducing conditions and in the presence of metallic surfaces, certain dissolved chlorinated organic compounds in groundwater will degrade to non-toxic compounds such as ethene, ethane, and chloride (Gillham and O'Hannesin, 1994, Gillham, 1996). The process appears to be abiotic reductive dehalogenation, with the metal serving to lower the Eh in solution and serving as an electron source in the reaction. Using iron as the reactive metal, reaction half-lives (the time required to degrade one half of the original contaminant mass) are commonly several orders of magnitude lower than those measured under natural conditions. The technology appears to have great promise for the remediation of contaminated groundwater because of these high rates of degradation, because iron is relatively inexpensive and the process requires no external energy supply, and because the compounds are degraded with production of very little hazardous (chlorinated) organic by-products.

Concurrent research has been conducted to evaluate the use of iron based systems to remove trace metal contaminants from groundwater. Metals which have been shown to be removed include chromium and nickel (Blowes et al, 1995, Puls et al, 1995), strontium (Marozas, 1996) and technicium (Clausen et al, 1995)

Field applications of iron based permeable treatment walls have been undertaken to remediate VOC and trace metal plumes. There are currently 10 installed in-situ systems removing VOCs (mainly TCE) from groundwater, and one system treating a combined VOC and trace metals plume. Field trials have also been conducted at U.S. DOE facilities and mine tailing sites in Canada to demonstrate trace metals removal using this technology.

Recent laboratory research has shown that the presence of another metal with iron may greatly enhance the degradation rates of VOCs relative to those obtained with iron alone. These materials may therefore decrease the amount of reactive material required in a given situation Various bimetallic media have been investigated, including palladium/iron, copper/iron, and nickel/iron. The University of Waterloo has shown considerable success with nickel/iron media in preliminary laboratory experiments (Gillham et al., 1997). The nickel/iron media will be referred to herein as enhanced iron.

In certain instances, periodic replacement of the reactive materials may be necessary to

rejuvenate the reactive surface and/or remove the accumulated trace metals from the medium. In these cases, a low density, more easily handled reactive material relative to granular metallic iron is desirable to facilitate these periodic operation and maintenance activities. Low density granular iron foams developed by Cercona Inc. of Dayton, Ohio have the potential to concurrently remove both VOCs and trace metals from solution.

Both the enhanced iron and iron foam materials will be evaluated as part of this study, in addition to commercial granular iron. The reasons for their inclusion are outlined in the next section.

The following uncertainties need to be investigated to facilitate field implementation of permeable treatment systems at any site

- 1) The dimensions and composition of treatment systems needed, based on the degradation rate of VOCs and removal rates of trace metals which would be obtained in site groundwater at the velocity which would occur in an *in-situ* or *ex-situ* treatment section at the site
- 11) The production and subsequent degradation rates of chlorinated compounds produced from the VOCs originally present in the site groundwater (e.g., dichloroethene isomers and vinyl chloride from TCE) These also affect treatment section dimensions
- 111) The amount of VOC degradation and trace metal removal possible versus required cleanup standards
- the potential for mineral precipitation Mineral precipitation rates affect long-term operations and maintenance requirements of the treatment system

We routinely evaluate these parameters in the course of field applications involving "conventional" granular iron, but they are even more important when examining the recently developed enhanced iron and iron foams

1.2 Site-Specific Applicability

A demonstration of the applicability of reactive barrier technologies will be conducted at the RFETS as part of a Department of Energy (DOE) EM-50 Subsurface Contaminants Focus Group sponsored program to identify cost-effective technologies for treating contaminated groundwater at DOE sites Laboratory bench scale testing is required to identify, test and develop suitable reactive materials for the demonstration of the applicability of reactive barrier technologies to treat VOCs in three contaminated groundwater plumes at RFETS. The site identified for initial testing is surface water seep SW059 which is at the distal end of the Mound Plume Water from SW059 is currently collected and treated in Building 891 at RFETS ETI has commercialized a patented method for metal-enhanced abiotic degradation of chlorinated organic compounds which could be used for treating VOCs not only in the SW059 plume, but also those present in the east trench plume and in 903Pad/Ryan's Pit plume. The system for the SW059 seep may also need to remove trace metals and radionvelides and may be designed to incorporate a removable "cassette" type of media container to facilitate periodic operations and maintenance (O&M) requirements The nickel iron material may produce rapid enough degradation so that only small quantities of this material are needed, facilitating the cassette design Alternatively, a low density foam could also be used in a removable cassette/reactor

A meeting was held in December 1996 at RFETS to discuss the planned testing program Based on this meeting, it was decided to evaluate the three types of reactive material described above, metallic iron, bimetallic material (nickel and iron), and iron foam pellets developed by Cercona Inc. It was originally planned to test an iron-zeolite foam pellet, however this material is in a very early stage of development. A foam composed of iron plus an iron alloy (an "HSI" foam) will be tested as the third medium, as tests by Oak Ridge National Laboratories (ORNL) and the University of Arizona have indicated this material to have the potential to remove both trace metals and VOCs. Rationale supporting this modification is presented in Appendix A. The tests have been designed so the results can be applied to remediation at all three plumes, as outlined below. Lastly, the tests have been scheduled to be completed by mid April, assuming a March 1 start date. This will facilitate a meeting at the RFETS in late April to discuss the possible installation of the system at SW-059.

2.0 SCOPE OF WORK AND OBJECTIVES

2.1 Bench Scale Study Objectives

The general objective of the Bench Scale Study is to examine VOC degradation rates in different media types to identify suitable materials for use in reactive barrier technologies at RFETS. It is our understanding that Sandia National Laboratories (SNL) will be examining suitability of iron and iron foam for the removal of trace metal and radionuclides in parallel tests

The degradation rate constants calculated from the laboratory data, and the measured changes in inorganic geochemical parameters, will be used to develop capital costs and potential O&M requirements for field-scale systems for the three plumes

2.2 Experimental Design

Uncontaminated groundwater obtained from a well on-site will be spiked and pumped at a constant rate into three columns containing reactive media. Uncontaminated groundwater will be shipped from the Site to the U of W laboratory. Based on historical concentrations, laboratory grade VOCs will be added to the groundwater to create the following approximate concentrations in the column influent,

VOC	Influent Concentration (ug/L)	Segment 5 Action Levels (ug/L)	Plume In Which VOC Is Present
PCE	4000	5	(all)
TCE	4000	5	(all)
cDCE	50	70	(SW059,east trench)
1,1-DCE	350	7	(all)
VC	50	2	(possible breakdown product)

<u>VOC</u>	Influent Concentration (ug/L)	Segment 5 Action Levels (ug/L)	Plume In Which VOC Is Present
CTET	850	5	(all)
TCM	55	8	(all)
DCM	50	5	(all)
1,1,1-TCA	25	200	(SW059,
			east trench)

These concentrations, close to historical maximums at the potential treatment locations, allow for a realistic assessment of the ability of the technology to treat field-scale VOC levels in each of the three plumes

Three reactive materials will be tested

- 1) one column of a commercial 100% iron source
- 11) one column containing 100% of the enhanced iron
- iii) one column containing granular iron HSI foam

The concentration of VOCs in groundwater will be monitored along the length of each column and monitoring continued until a steady-state concentration profile develops in each column. An initial flow rate of about 1.5 pore volumes/day will be used in all three columns. This rate corresponds to a flow velocity of about 2 to 3 ft/day, which is considered a reasonable approximation of field conditions. It will also allow us to complete the test in the required time period. Based on our experience, about 30 to 50 pore volumes of flow will be needed in the iron (and iron foam) materials to reach "steady-state" conditions (where concentrations at a point along the column remain stable over time). At this flow rate, 30 to 50 pore volumes will have passed through the columns in about three to five weeks.

While our experience with the enhanced iron is more limited, it has shown that several additional pore volumes of flow may be required to reach these steady state conditions, depending on site groundwater chemistry. If the enhanced iron column has not reach steady state by about 40 pore volumes, the flow rate in this column will be increased up to 3 pore

volumes/day for another 25 to 50 pore volumes This increased flow rate will allow the enhanced iron test to be completed within the prescribed time frame

Details concerning column test methodology and interpretation of these VOC profiles and related test data are presented in the following section. The most significant results of these column tests are the VOC degradation rates, which will be used to evaluate the field applicability of the technology in terms of the specific objectives outlined in Section 2.1 Existing data from other tests of these materials can be used in this evaluation.

No control column will be operated for this study Several previous tests have shown that there is no effect of the plexiglass columns or pump tubing on observed concentrations

3.0 STUDY METHODS

This section describes the methods to be used to conduct the study and present and interpret the data from this study. Analytical and QA/QC procedures are summarized herein and are presented in more detail in Appendix B. The study will be conducted by the Institute for Groundwater Research, University of Waterloo (U of W) with input from ETI on the design and monitoring of the test. The laboratory's Health and Safety Plan is contained in Appendix C.

3.1 Groundwater Shipment and Storage

Groundwater from the site will be shipped to U of W, for use in the study Chain of custody forms received with the samples will be appended to our report. Samples will be catalogued and their use recorded as shown in Table 1. Reagent grade organic chemicals will be added to the groundwater to simulate the concentrations expected, as outlined in Section 2.2.

3.2 Column Equipment and Materials

Table 2 presents a list of analytical equipment which will be employed during this study. All equipment has been set up and is maintained in strict accordance with manufacturer's specifications.

The 50 cm long, 3 8 cm inside diameter (ID) plexiglas columns (Figure 1) used in this study are equipped with several sampling ports along the length of each column at distances of 2 5, 5, 10, 15, 20, 30 and 40 cm from the inlet end as well as the influent and effluent. Each sampling port consists of a nylon Swagelok fitting (0 16 cm) tapped into the column side, with a sterilized syringe needle (0 12 cm outside diameter (OD) x 0 17 cm ID x 3 8 cm long) placed inside. To prevent column packing materials from entering the needle, glass wool will be placed in the barrel. This sampling port allows the sample to be collected from the midpoint of the column. Each syringe needle is fitted with a Luer-Lok fitting, to which a syringe may be attached to collect a sample. The sampling ports are sealed by a Luer-Lok plug which is twisted snugly into the syringe needle between sampling events.

The commercial iron used will be -8 to +40 mesh obtained from Connelly GPM Inc (CON) The CON iron has been used in several treatability tests and a field application. Based on laboratory results, it represents the most reactive iron of the three available iron sources. CON iron will also be used as the basis for the enhanced iron. Foamed HSI iron pellets will be obtained from Cercona Inc.

To fill the columns with reactive material, aliquots of material are packed in vertical lifts within the columns. Each column is packed to assure a homogeneous mixture. Each lift is tamped down with a flat metal rod. Care is taken to avoid layering, by scarifying the top of each lift before applying the next lift. Average bulk densities, porosity, and pore volume measurements are determined gravimetrically. All column experiments are performed at room temperature.

Groundwater will be transferred from the shipping containers into a Teflon bag with no headspace, and spiked with VOCs to the concentration specified in Section 2.2. This influent solution is injected into the lower (influent) end of the column at a constant volumetric flow rate corresponding to a flow velocity of 2 to 3 ft/day using a peristaltic pump (Ismatec IPN). The pump tubing is Viton tubing, and all other tubing used to connect system components is Teflon (1/8 inch OD x 1/16 inch ID).

3.3 Data Collection (Sampling Protocols)

As discussed previously, the columns are sampled over time until a steady-state profile is achieved (i.e., when concentrations at a point in the column remain relatively unchanged over time). After removing a flush volume from the sampling port needle, 2.0 mL to 3.0 mL samples are collected for analyses for VOCs from each port. Groundwater samples are also collected from the influent and from the effluent overflow bottle.

Chlorinated ethene CTET, TCM, and 111-TCA concentrations will be monitored at the inlet, outlet, and sampling ports of each column to determine when steady state has been reached at the flow rate of \pm 15 pore volumes/day. These profiles will be collected every 5 to 10 pore volumes. As steady state conditions are approached, analyses of DCM will be completed in addition to the above VOCs. Periodic Eh and pH profiles will also be collected during this time. Flow rates are measured initially using a graduated cylinder and stopwatch, then volumetric flow measurements are made concurrently with each profile

We will also collect and analyze the column influent and a minimum of two effluent (outlet) samples from each column for major cations (Ca+2, Mg+2, Na+, Fe+2, Mn+2, K+, Ni+2), major anions (SO4-2, Cl-, NO₃-, NO₂-) and carbonate and bicarbonate alkalinity

Previous experience has shown that it is very difficult to obtain accurate measurements of dissolved oxygen from the column, Eh is measured in its place. The design of the laboratory columns does not permit the accurate measurements of hydrogen gas evolved during the corrosion reaction, or the accurate measurement of non-chlorinated hydrocarbon gases (ethene, ethane) which represent the end products of the dechlorination reaction. The main use of the hydrocarbon gases has been to provide evidence of dechlorination (MacKenzie et al., 1995, Gillham, 1996). There is sufficient data present from these and other previous studies that modification of column design to allow confirmation of dechlorination here using these gases was not considered cost effective.

3.4 Analytical Protocols

The analytical protocols employed by the U of W for organic and inorganic analyses are described in detail in Appendix B In summary, the following methods will be used

- select VOCs will be analyzed using micro-extraction techniques (a modification of the method of Henderson et al, 1976) and gas chromatography using an election capture detector or using headspace analysis techniques and gas chromatography equipped within a photoionization detector,
- pH and Eh using reference electrodes,
- dissolved cations using inductively coupled plasma methods,
- anions using ion chromatography or colorimetry, and
- alkalinity using a titration method

QA/QC procedures are described in detail in Appendix B Recoveries of spiked blanks, instrument calibration range vs sample range, and method blanks will be presented in the laboratory test report (Section 5) Method detection limits for organic and inorganic compounds are shown in Tables 3 and 4 respectively

3.5 Column Data Presentation and Interpretation

For each test column VOC concentration will be plotted as a function of distance along the column after steady-state conditions have been achieved. The flow rate will be used to calculate the residence time at each sampling position for each profile. The results of the column tests are plotted as concentration of the VOCs in $\mu g/L$ versus residence time in hours. Graphs will be prepared illustrating these results. Degradation rate constants will be calculated for each organic compound originally present in the groundwater, using the first-order kinetic model

$$C = C_0 e^{-kt}$$
 (1)

where

VOC concentration in solution at time t,

 C_0 = initial VOC concentration of the influent solution,

k = first order rate constant, and

t = time

By rearranging and taking the natural log, equation (1) becomes

$$ln (C/C_0) = -kt$$
(2)

The time at which the initial concentration declines by one-half, $(C/C_0 = 0.5)$, is the half-life, which, by rearranging equation (2), is given by

$$t_{1/2} = 0.693 / k$$
 (3)

The decay constants k [1/time], will be computed from the slope of the first-order model, obtained by fitting equation (2) to the experimental data Half-lives, along with corresponding

correlation coefficients (r^2) values will be provided. The r^2 values indicate how well the first-order model fits the experimental data

A similar approach will be used for quantifying the observed degradation of any chlorinated breakdown product. The peak concentration of the breakdown product will be used as C_0 in these calculations

Plots of Eh, pH and concentrations of degradation products versus column distance (residence time) will be made for each test

Inorganic chemical results from the column tests will be reviewed and interpreted to evaluate the possible extent of mineral precipitates on field-scale technology application. Changes in dissolved calcium, iron, and alkalinity between column influent and effluent samples are of particular interest when examining the potential for carbonate and/or hydroxide mineral precipitation. Preliminary estimates of clogging rates which might occur in the reactive media will be made from these data. A geochemical model such as MINTEQA2 (US EPA, 1991) may be used to assist in this interpretation, by calculating saturation indices of the carbonate and hydroxide minerals of concern

3.6 Disposal of Residuals

All residuals (effluent groundwater and column materials) will be disposed of according to appropriate Canadian and Ontario environmental regulations

4.0 REPORTING

A Report will be prepared after the column tests have been completed to address the objectives of these studies as outlined in this work plan

The report will contain the following topics

- any deviations from this work plan and their rationale will be described,
- test design, test procedures, and sampling and analytical protocols will be summarized,
- QA/QC data will be summarized as they affect the validity of test results,
- VOC degradation rates from both columns will be tabulated for each type of reactive
 material tested. The production and subsequent degradation of chlorinated products will
 also be quantitatively discussed. The ability of the process to meet the Performance
 Criteria will be described,
- discussions of long-term performance based on the data evaluation described in the previous section, and
- a conceptual design for scaleup and capital and O & M costs for a full-scale system

Field residence time requirements to reach Tier II Action Levels for each plume and each media type will be evaluated. The residence time affects the dimensions (and cost) of the field-scale system.

The residence time will be calculated using a model developed by ETI staff that incorporates the concurrent production and degradation of the various organics present in the groundwater to calculate the cumulative residence time required,

• Key operational aspects of the field-scale system will also be addressed. These include the potential effects of mineral precipitates on system performance (i.e., plugging and/or loss of reactivity) and the expected lifetime of the iron using published corrosion rates (Reardon, 1995) and ongoing tests

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5.0 SCHEDULE - BENCH SCALE STUDIES

Our schedule for the various activities is given below. It allows the study to be completed by mid-April, 1997. Those time periods assume an award of contract in late January 1997.

Test Plan

Draft Submitted

Final Submitted

March 7, 1997

Column Tests

March 4 - April 18, 1997 (±6 wks)

February 26, 1997

Data Interpretation, Submission of Draft Report

April 7 - April 25, 1997

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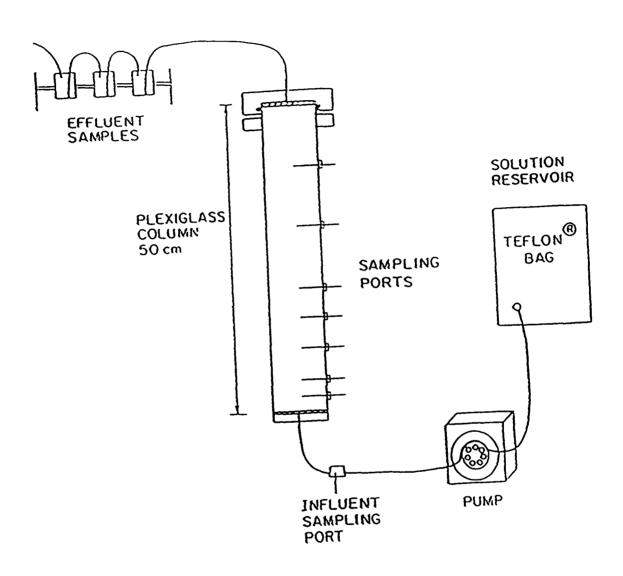


Figure 1. Schematic of the apparatus used in the column experiments

TABLE 1:	RECORD OF SAMPLE STORAGE

EXPERIMENT	NO:	

<u> </u>	<u></u>				
U of W	NUMBER OR	SIZE	DATE	REASON OR	COMMENT
NUMBER	WELL NO	OF	OPENED	OPENING	
ASSIGNED		GLASS			
1 HOUTON LED	BOTTLE	BOTTLE			
	BOTTLE	BOTTLE			

TABLE 2: EQUIPMENT LIST

Organic Analyses

Gas Chromatographs

Hewlett Packard 5890 Series II gas chromatograph with Autosampler HP 7673 Injector and HP 3396C Series II Integrator

Ni⁶³ electron capture detector (ECD) DB-624 capillary column Makeup gas was 5% methane and 95% argon Carrier gas Helium

Hewlett Packard 5890 Series II gas chromatograph with a HP 3396B Series II Integrator

HNU Photoionization (PID) Detector Bulb ionization potential of 10.2 eV NSW-Plot capillary column (15 m x 0 53 mm ID) Carrier gas Helium

Inorganic Analyses

Cation

Inductively coupled plasma, Thermo Jarrell Ash, Model Iris

Anion

Ion Chromatograph, Dionex Model 2000

Eh/pH

Markson meter, Model 90

- Redox Potential (Eh) Electrode, Ag/AgCl Reference

- pH/Reference Combination Electrode

Other Laboratory Equipment

Balance, Mettler Analytical AE163, 5 Decimal Balance, Mettler PJ3000, 2 Decimal Laboratory Pump - Ismatec, 4 channel Oven, Boekel, Model 107801 Shaker, Labline Orbit, Model 3520 Stopwatches Teflon Bag

eof//Sept 1996

TABLE 3: METHOD DETECTION LIMITS - ORGANIC COMPOUNDS

Compound	MDL (µg/L)
Tetrachloroethene (PCE)	18
Trichloroethene (TCE)	1 6
1,1-Dichloroethene (11DCE)	3 2
cis-1,2-Dichloroethene (cDCE)	7 8
trans-1,2-Dichloroethene (tDCE)	1 9
Vınyl Chloride (VC)	0 7
Carbon Tetrachloride (CTET)	1 0
Trichloromethane (TCM)	1 2
Dichloromethane (DCM)	3 9
1,1,1-Trichloroethane (TCA)	1 1

TABLE 4: DETECTION LIMITS (DL) - INORGANIC COMPOUNDS

Inorganic Compounds	DL (mg/L)
Cations	
Iron (Fe)	0 02
Sodium (Na)	0 1
Magnesium (Mg)	0 1
Calcium (Ca)	0 1
Potassium (K)	0 5
Manganese (Mn)	0 01
Nickel (Ni)	0 01
Chromium (Cr)	0 005
Cadmium (Cd)	0 003
Zinc (Zn)	0 005
Anions	
Chloride (Cl)	1 0
Sulphate (SO ₄)	2 0
Nitrate (as N)	0 05
Ammonia (as N)	0 05
Alkalınıty (as CaCO ₃)	1 0

Appendix A
Rationale for Selection of Iron Foam

ETI MEMO

To John Hopkins, Rocky Mountain From John Vogan, ETI Remediation Services Ref Selection of Candidate Material for Column Testing 12 February 1997 Date RFETS Treatability Studies-31212 88

As you are aware, three candidate materials have been identified for column testing as part of our upcoming treatability study using groundwater from the RFETS These three were commercial granular iron, bimetallic nickle-iron media, and a zeolite-iron mixture Both Dr Dianne Marozas of SNL and I have recently talked with Dr Robert Bowman of New Mexico Tech regarding his work with the pelletized zeolite-iron mixtures. There appears to be some uncertainty regarding the optimum composition of this material for combined VOC and trace metal/radionuclide removal I understand that Dr Bowman is still investigating different zeolite-iron mixtures, with and without surfactant coatings

Given the time frame of this study, Dianne Marozas suggested that one of the iron foam materials produced by Cercona Inc could be tested in place of the zeolite-iron pellets This foam has been shown to be capable of trace metal and radionuclide removal (adsorption) by various DOE investigators, including Dr Maroza's group Both the University of Waterloo and Oak Ridge National Laboratories have conducted preliminary studies which indicated that VOC degradation at reasonable rates occurred in these materials. Given its low density, the iron foam certainly represents a possible candidate material for use in a "removable" treatment canister/cassette for the SW-59 Seep system

Based on the above, we suggest testing this material in the third column in place of the zeoliteiron material in the column studies. We can provide references concerning the prior tests of the iron foams in our work plan, which will be submitted following our notification to proceed gol V. with this project If you have any comments, please call me

Dr Dianne Marozas, SNL ∞ Stephanie OHannesin, University of Waterloo Jennifer Uhland, Kaiser-Hill

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ETI MEMO

То	John Hopkins, RMRS	From	John Vogan, ETI
Ref	Selection of Iron Foam Material for Use in RFETS Column Studies - 31212 10	Date	24 February 1997

Further to our recent memorandum regarding the use of Cercona Inc iron foam material in RFETS column studies, I spoke to Rich Helferich, president of Cercona, regarding the most appropriate material for use in these tests. A summary of our discussions follows

There are two types of iron foam material that have undergone previous laboratory and/or field testing. The first is a pelletized iron foam, which has been shown to degrade VOCs in University of Waterloo (U of W) batch tests, and remove strontium and radionuclides in DOE (SNL, ORNL) tests. Cercona has also developed a bimetallic iron foam, which Rich Helferich refers to as an HSI material. The two metals used in this case are iron and a high silica iron alloy. This material has not been tested by U of W or SNL but has been shown to remove uranium in ORNL batch tests. It also promoted faster rates of VOC degradation than the pelletized iron foam in these ORNL tests. The HSI material also has been tested in longer term column studies at the University of Arizona, where it has reportedly outperformed one of the three identified commercial iron sources (Master Builders Iron)

Based on these test results, Rich Helferich's recommended that the HSI material be used in the RFETS testing program. From our perspective, we see no reason to disagree with recommendation. Providing the rest of the study team concurs, we will obtain this material from Cercona in the next few days for use in the column tests.

JV/cf

cc Dr D Marozas, SNL
S. O'Hannesin, U of W
R. Helferich, Cercona
J Uhland, Kaiser Hill

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APPENDIX B

LABORATORY SAMPLING AND ANALYSES FOR TREATABILITY STUDIES OF THE ENVIROMETAL PROCESS

Appendix B

Laboratory Sampling and Analyses for Bench Scale Studies involving the EnviroMetal Process

> Institute for Groundwater Research University of Waterloo Waterloo, Ontario, Canada N2L 3G1

B1.0 Introduction

The organic compounds in groundwater received by the University of Waterloo laboratory for treatability testing have already been characterized by commercial laboratories using standard EPA analytical methods involving routine techniques such as GC/MS and/or GC purge and trap. Since the compounds in the groundwater have been well defined, then more specific sample analyses is conducted during the treatability test to confirm initial concentrations and to evaluate the degradation process by tracking the presence or absence of compounds in the groundwater. These particular analyses no longer require GC/MS or GC purge and trap methods, since these methods are costly, require large amounts of groundwater and are too time consuming to be used in the treatability tests. Separatory funnel or continuous solvent extraction techniques are also slow and labour intensive, and may also suffer from volatilization losses. Specific analytical procedures are used for treatability testing. The methods are described below.

The organic research laboratory at the University of Waterloo is well-equipped for analysis of chlorinated organic compounds, and the two senior staff members each have close to ten years of experience with these types of analyses Previous interlaboratory comparisons have confirmed the quality of data from the laboratory Though standard research analytical procedures are used, the laboratory is not EPA certified

B2.0 Receipt and storage of Groundwater

Upon arrival of shipped samples at the U of W laboratory, the shipping containers (coolers) will be examined for integrity and samples will be inspected for cracks, breakage, leakage, inverted septa, inappropriate caps, air bubbles and appropriate labelling as per sample shipment forms. All samples received will be logged on a sample submission form as shown in Table A1. Prior to their use in the bench scale studies, all samples will be stored in a secure refrigerator at 4°C. The use of these samples in the bench scale studies will be logged on the record of sample storage form

shown in Table A2

Samples of groundwater received at the University will be analyzed immediately upon arrival for baseline VOCs of concern, using the methods described below

B3.0 Organic Analytical Procedures

B3.1 VOCs via Pentane Micro-Extraction

The less volatile halogenated organics such as tetrachloroethene (PCE) and trichloroethene (TCE) are extracted from the aqueous phase using pentane micro-extraction technique. The extraction from the aqueous phase uses a methodology derived from a pentane liquid-liquid extraction (US EPA, 1976; Henderson et al., 1976, Glaze et al., 1981). This method is especially suited for bench scale studies, since it can accommodate small sample sizes

After removing a flush volume from the sampling port needle, a 2 0 mL aqueous phase sample is taken directly from the column using a 2 0 mL glass syringe. The organic analyses is performed immediately, and has no holding time associated with it. This sample is then transferred directly from the glass syringe to a Teflon®-faced septa screw cap vial containing 2 0 mL pentane with an internal standard of 1,2-dibromoethane, at a aqueous sample to pentane ratio of 2 0 to 2.0 mL. The sample is placed on a rotary shaker for 10 minutes to allow equilibration between the water and pentane phases. The pentane phase is removed and transferred to an autosampler vial for injection into the gas chromatograph.

Using a Hewlett Packard 7673 auto sampler, a 10 μ L aliquot of pentane with internal standard was automatically injected directly onto a Hewlett Packard 5890 Series II gas chromatograph. The chromatograph was equipped with a Ni⁶³ electron capture detector (ECD) and DB-624 megabore capillary column (30 m x 0 538 mm ID, film thickness 3 μ m). The gas chromatograph has an initial temperature of 50°C, with a temperature time program of 15°C/minute reaching a final temperature of 150°C. The detector temperature is 300°C. The carrier gas is helium and the makeup gas is 5% methane and 95% argon, at a flow rate of 30 mL/min

B3.2 VOCs via Headspace Analysis

For the more volatile compounds such as cis 1,2-dichloroethene (cDCE), trans 1,2-dichloroethene (tDCE), 1,1-dichloroethene (11DCE) and vinyl chloride (VC) a headspace analysis is conducted A 2.5 mL aqueous phase sample is taken directly from the column using a 3.0 mL glass syringe This sample is then transferred directly to a Teflon®-faced septa screw cap vial, creating a

headspace with a ratio of 2.5 mL headspace to 2.5 mL aqueous sample. The samples are placed on a rotary shaker for 15 minutes to allow equilibration between the water phase and gas phase. For analysis, a 500 µL gas sample is injected directly onto a Hewlett Packard 5890 Series II gas chromatograph. The chromatograph is equipped with a HNU photoionization detector (PID) with a bulb ionization potential of 10 2 eV. The gas chromatograph is fitted with a fused silica capillary NSW-PLOT column (15 m x 0.53 mm ID). An isothermal oven temperature of 160°C and a detector temperature of 120°C are used. The carrier gas is helium with a flow rate of 4.1 mL/min

B3.3 Reagents

The following reagents are used glass distilled pentane, spectranalysed methanol, organic-free distilled water, reagent grade chemicals, such as tetrachloroethene (PCE), trichloroethene (TCE), 1,2-dibromoethane (EDB) [internal standard], 1,1-dichloroethene (1,1-DCE), cis 1,2-dichloroethene (cDCE), trans 1,2-dichloroethene (tDCE) and vinyl chloride (VC)

B3.4 Sample Bottle Preparation

Bottles and other glassware are soaked in a commercial alkaline cleaning solution for 24 hrs, then rinsed with tap water, 10% nitric acid, tap water and a final rinse of distilled water. The glassware is baked overnight for 24 hrs at 110°C. Upon cooling, the glassware are covered with foil. The Teflon®-faced silicon septa are boiled in distilled water for one hour and then baked at 110°C for two hrs.

B3.5 Quality Control and Quality Assurance

Samples and standards are equilibrated to room temperature (approximately 22°C) before extraction. All solutions are prepared gravimetrically. A stock standard is prepared by injecting the various pure organic compounds through a septum into an appropriate aliquot of spectranalysed methanol. These stock standards are stored at 4°C when not in use. Stock standards are prepared fresh as often as required by their stability, and will be checked regularly for signs of deterioration (ie, discoloration, formations of precipitates, and changes in concentration) and shall be discarded if signs of deterioration are evident (Section 3 5 2)

B3.5.1 Machine Calibrations

Using a stock standard, a set of five calibration standards are made up in organic-free water, over a concentration range of 10 to 2500 μ g/L for the micro-extraction samples on the ECD gas

chromatograph and 10 to 1000 μ g/L for headspace analyses on an PID gas chromatograph. The instrument is calibrated for all target compounds creating a multi-point calibration curve which is stored within each instrument. Calibrations have a limited lifetime because instrument sensitivity drifts and recalibration becomes necessary to maintain the accuracy of the analysis (Hewlett Packard, 1990). Calibration curves are produced and evaluated on a daily basis to ensure that the system is within calibration. If the percent recovery of the daily checks are consistently greater than 110% or consistently less than 90% the system is recalibrated.

If sample concentrations are not within the calibrated concentration range, dilutions are performed depending on sample concentration

B3.5.2 Analytical Accuracy

Each machine calibration is checked daily. The analytical accuracy involves spiked blanks which are spiked with VOCs from laboratory grade reagents (a different stock standard than that which the calibration was derived), are made up daily in order to check, the machine calibration and analytical method. These are recorded as percent recoveries (%R). Method blanks are carried throughout the entire analytical procedure routinely. Two blanks and 10 to 15 spiked blanks are analysed every time sampling is undertaken. These quality control and assurance procedures closely follow those given in US EPA (1986).

Duplicate sample profiles are taken on one occasion for each column, in order to assure that the sampling procedure is reproducible, and duplicate analyses were performed on these samples in order to evaluate the reproducibility of the analytical procedures

B3.6 Method Detection Limits

An aliquot of a laboratory standard (VOC of interest) is added to organic free water, stirred and then a minium of ten samples are transferred into analytical bottles and analysed accordingly Analyses is conducted in the same manner as handling a typical sample on each type of analytical equipment, depending on the methodology

Method detection limits are determined from recent and ongoing laboratory studies for each compound as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. The method detection limits are determined from analysis of samples from a solution matrix containing the analytes of interest. Detection limits for all compounds studied are determined using the EPA procedure for Method Detection Limit (MDL) (US EPA, 1982)

B4.0 Inorganic Analyses

B4.1 Redox Potential (Eh) Measurements

Redox potential (Eh) is determined using a combination Ag/AgCl reference electrode with a platinum button and a MarksonTM Model 90 meter The electrode is standardized with ZoBellTM solution Millivolt readings were converted to Eh, using the electrode reading and the standard potential of the Ag/AgCl electrode at the measurement temperature

The Eh electrode is polished daily and checked with the standard Zobell solution A 2.5 mL aqueous sample is removed from each sampling port and placed in a 5 mL glass vial and analysed immediately. The electrode is rinsed thoroughly with distilled water, wiped clean and is suspended in the sample. The electrode is allowed to equilibrate in the sample and a reading is taken. The temperature is also recorded.

B4.2 pH Measurements

The pH measurements are conducted using a combination pH/reference electrode and a MarksonTM Model 90 meter, standardized with the commercially available buffers 7 0 and the appropriate buffer of either 4 or 10. The electrode is standardized with the appropriate buffer and rinsed thoroughly with distilled water, wiped clean and then placed in the sample. A 2.5 mL aqueous sample is removed from each sampling port and placed in a 5 mL glass vial and analysed immediately.

B4.3 Cations and Anions

Aqueous water samples are collected from the influent and effluent of each column and sent to MDS Environmental Services Ltd, 6850 Goreway Drive, Mississauga, Ontario for cation and anion analyses. Cation analyses include Fe (total), Na, Mg, Mn, Ca, K and a suite of other cations. These analyses are performed using inductively coupled plasma (ICP). The unfiltered, 60 mL samples were acidified to a pH of 2 with nitric acid and stored at 4°C until analysed. Anion analyses, including Cl, SO₄ and others are performed on 60 mL unfiltered samples using ion chromatography. Alkalinity is determined by automated colorimetry. Detection limits for the inorganic parameters are included in Table 2.

B5.0 References

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APPENDIX C LABORATORY HEALTH AND SAFETY PLAN (HSP)

APPENDIX C

Laboratory Health and Safety Plan (HSP)

1.0 Introduction

This health and safety plan (HSP) has been prepared in accordance with the information contained in the document entitled "Guideline for Conducting Treatability Studies Under CERCLA" (Interim Final, December 1989) All laboratory tests conducted during this treatability study will be performed at the Earth Sciences Department, University of Waterloo (U of W), Waterloo, Ontario, Canada

Health and Safety practices at the U of W are governed by legislation passed under the Province of Ontario Occupational Health and Safety Act (Revised Statutes of Ontario, 1990) This act sets out the rights and duties of all parties in the workplace, establishes procedures for dealing with workplace hazards, and provides for enforcement of regulations passed under the Act. The other major piece of legislation governing the University's health and safety practices is the Workplace Hazardous Materials Information System (WHMIS), which mandates that workers be informed about hazards in the workplace and receive appropriate training to enable them to work safely. WHMIS is legislated jointly through the federal (Canadian) and provincial (Ontario) governments. Radiation laboratories are inspected three times each year.

The U of W Department of Health and Safety is responsible for implementation of appropriate health and safety regulations in all laboratories on campus A Health and Safety Committee inspects all laboratories at least once each year to ensure compliance with relevant guidelines

2.0 Hazard Analysis

Groundwater used in the treatability studies will be spiked with dissolved volatile organic compounds at concentrations in the ppb to ppm range. The reactive solid phase material to be used in the study, namely metallic iron and nickel, is non-hazardous. Given the above, there are no specialized health and safety practices which need to be applied during the treatability tests other than those routinely followed in the U of W laboratories. These procedures are described in detail in the following sections.

3.0 Employee Training

At a minimum, all laboratory personnel involved in this project have completed the WHMIS training session prepared by the U of W Health and Safety Department This training session covers the following topics

- a review of relevant legislation,
- appropriate safety precautions in handling hazardous materials, including instruction regarding Material Safety Data Sheets (MSDSs) and labelling of laboratory supplies,
- appropriate work practices and hygiene practices;
- chemical spill procedures, and
- waste disposal

In addition, laboratory personnel performing the majority of tasks in this study have completed the 40 hour certification requirements under U S Department of Labour, Occupational Safety and Health Administration in compliance with SARA Title III and 28 CFR 1910, updated annually with an 8 hour refresher course. All personnel working with radioisotopes have successfully completed the University of Waterloo Radiation Training Course, and are designated as atomic radiation workers. Resumes for laboratory personnel are contained in Appendix A of this document.

4.0 Health and Safety Personnel

Stephanie O'Hannesin, a research coordinator in the University of Waterloo Earth Sciences Department serves as the Health and Safety representative for the laboratory where the treatability studies are completed Ms O'Hannesin is responsible for completion of the laboratory tests in this study. Her health and safety responsibilities include the following.

- ensuring that all new laboratory personnel complete appropriate training sessions,
- ensuring that MSDSs are provided for substances used in the laboratory, and maintaining an inventory of these substances,
- ensuring that proper storage, labelling and waste disposal procedures are followed in the laboratory,
- ensuring all material handling licences are kept up to date, and
- serving on the University Radiation Safety Committee

5.0 Protective Equipment

The laboratories at the U of W involved in this study are equipped with the following protective equipment

- fumehoods,
- emergency eye wash and shower stations,
- fire extinguishers and alarms,
- sorbents for liquid spill clean-up, and
- spill clean-up procedures, names and phone numbers of contact persons, and emergency exits are clearly posted in each laboratory

The following personal protective equipment is used as required

- laboratory coats,
- non-adsorbent and disposable gloves,
- protective eye glasses, and
- dust and organic vapour filter masks

6.0 Monitoring Activities

The concentrations of dissolved VOCs in groundwater to be used in the laboratory and the experimental methods used do not necessitate the use of personal respiratory monitoring equipment by laboratory personnel. The possible presence of organic vapours in the laboratory is monitored when necessary depending on the nature of the laboratory experiment.

7.0 Residuals (Waste) Management

The residuals produced during the treatability study consists of groundwater and reactive media used in the laboratory experiments. These are disposed of using waste handling facilities at the U of W in accordance with all relevant Province of Ontario environmental regulations.

8.0 Chemical Spill Procedures

The following chemical spills procedures are employed at the U of W laboratories in accordance with WHMIS requirements

- proper personal protective equipment (e g, gloves, respirators) is worn as specified in the MSDSs,
- liquids are absorbed or neutralized,
- solids are swept into a container,
- the area is ventilated to dispel vapours if required,
- the affected area is washed with soap and water or detergent, and
- all materials used in cleanup, including absorbed liquids are disposed of as hazardous waste. The waste is labelled appropriately

If proper equipment is not immediately available to safely contain and decontaminate a spill, the area is evacuated and the appropriate personnel contacted

CURRICULA VITAE OF LABORATORY STAFF

curriculum vitae

October 1993

Stephanie O'Hannesin

Institute for Groundwater Research University of Waterloo Waterloo, Ontario N2L 3G1 Phone (519) 888-4567 Ext 3159 Fax (519) 746-7484

Education:

M.Sc.

Masters of Science - Hydrogeology

1993

Department of Earth Sciences,

University of Waterloo, Waterloo, Ontario

B.E.S 1981 Bachelor of Environmental Studies - Honours Geography,

Department of Geography,

University of Waterloo, Waterloo, Ontario

Professional Experience:

1988 - present

Institute for Groundwater Research

University of Waterloo, Waterloo, Ontario

Research Co-ordinator

- major responsibilities include design, coordination and supervision of contract research projects for government agencies and industrial clients
- establish work schedules based on contract requirements and deadlines
- administer internal and contract financial accounts
- proposal and report writing
- data compilation and analyses
- manage the following three laboratories:

low level radiation porous media hydrogeochemistry

- supervise staff and students

Stephanie O'Hannesin

October 1993

1985 - 1988

Institute for Groundwater Research University of Waterloo, Waterloo, Ontario Senior Research Technician

- executed and supervised experimental aspects of contracts
- assimilated data, preliminary interpretation and assisted in preparation of reports
- supervised three junior staff members
- performed various administrative responsibilities
- analytical capabilities included operation of gas chromatographs, scintillation counter and gamma counter (radiolabelled tracers)

1981 - 1985

Institute for Groundwater Research University of Waterloo, Waterloo, Ontario Hydrogeology Technician

- conducted various laboratory experiments
- operated general laboratory equipment and field equipment
- performed analyses for trace organics on gas chromatographs
- field sampled organic and morganic samples
- assisted in data compilation, interpretation and report preparation

Work Related Skills:

Technical Services Committee, Earth Science Department, University of Waterloo Radiation Safety Committee, University of Waterloo

OHSA (Occupational Health and Safety Administration Training) Course

- 40 Hour Course Feb 1992
- 8 Hour Refresher Feb 1993

WHMIS (Workplace Hazardous Materials Information System Training) Course, University of Waterloo

Radiation Safety Course, University of Waterloo

Computers: Familiar with Quattro Pro, Word Perfect, Windows, DOS

Extensive experience on:

Gas Chromatographs (ECD/FID)

Beta Counter (Liquid scintillation counting)

Gamma Counter

October 1993

Publications

Refereed Publications

Gillham R W. and O'Hannesin, S. F., 1993. Metal-enhanced degradation of halogenated aliphatic compounds (in submittal).

Lyngkilde, J, Christensen, T.H, Gillham, RW., Larsen T., Kjeldsen, P., Skov, B, Foverskov, A., O'Hannesin, S.F., 1992. Degradation of specific organic compounds in leachate polluted groundwater. Chapter 4.3 in Landfilling Of Waste. Leachate, Elsevier Applied Science, pp. 485 - 496

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Non Refereed Publications

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Stephante O'Hannesin October 1993

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Stephanie O'Hannesin October 1993

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Stephanie O'Hannesin

October 1993

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Gillham, R.W. and O'Hannesin, S.F., 1988 Sorption of soluble constituents of petroleum products on sampling-well materials (Poster) Petroleum Hydrocarbons and Organic Chemicals, Houston, Texas

(oral presentations underlined)

Additional Oral Presentations

1993	August 19	Westinghouse Hanford Company, Richland, WA
1992	June 11	General Electric Corporate Laboratories, Schenectady, NY
1991	June 25	University of Waterloo, Solvents in Groundwater Consortium
1988	Jan 25	University of Waterloo, Dept of Earth Sciences
1988	June 11	Danish Institute of Technology, Lyngby, Denmark
	May-June	Also demonstrated an <i>in situ</i> field testing device for biochemical reaction rates and retardation factors. (Invited)

Wayne L. Noble

Institute for Groundwater Research University of Waterloo Waterloo, Ontario N2L 3G1 Phone (519) 888-4567 Ext 3159 Fax (519) 746-7484

Education:

B.Sc. 1991

Bachelor of Science - Honours Applied Chemistry

Department of Chemistry,

University of Waterloo, Waterloo, Ontario

Professional Experience:

Sept 1992 -Present Institute For Groundwater Research, University of Waterloo

Waterloo, Ontario

Hydrogeological Technician

- Field sampling

- Trace organic and inorganic chemical sampling and analysis

May 91-Aug.92 Department of Earth Sciences, University of Waterloo

Waterloo, Ontario Research Technician

- involved in a chemical kinetic study of the oxidation of mine tailings

Work Related Skills:

- Familiar with most analytical equipment, GC, HPLC, AAS, ICP, FT-IR, XPS, & SEM
- Strong background in wet chemical analysis
- OSHA (Occupational Health and Safety Administration Training)
 40 Hour Course Sept 1993
- WHMIS Safety Course, University of Waterloo
- Computer literate in 'C', Pascal and Fortran
- Familiar with Lotus, Word Perfect, Windows and DOS

Greg Friday

Institute for Groundwater Research

University of Waterloo

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Phone (519) 888-4567 Ext 3159

Fax (519) 746-7484

Education:

B.Sc.

Bachelor of Science - General Science

1986

Science Department,

University of Waterloo, Waterloo, Ontario

Professional Experience:

1985 -

Institute for Groundwater Research,

Present

University of Waterloo, Waterloo, Ontario

Hydrogeological Technician

-Field sampling

-Routine chemical sampling and analysis of batch and column experiments

Oct 1984

Department of Earth Sciences,

- Apr 1985

University of Waterloo, Waterloo, Ontario

Research Technician

- involved in a research study of the diffusion rates of various radioactive

nuclides into several different concrete materials

Work Related Skills:

OSHA

-Occupational Health and Safety Administration Training

-40 Hour Course Feb 1992

-8 Hour Refresher Feb 1993

Radiation Safety Course, University of Waterloo, 1986 Spill Safety Course, University of Waterloo, 1992 Machine Shop Course, University of Waterloo WHIMS Safety Course, University of Waterloo

Computer work: Lotus, Word Perfect

Extensive Experience on:

- Field installation of monitoring wells and equipment
- Gas chromatography (FID, ECD, PID) of trace organics
- Fraction organic carbon analysis (FOC) by combustion
- Beta counter (Liquid scintillation counting)
- Gamma counter